(English Translation)

## PATENT OFFICE JAPANESE GOVERNMENT

This is to certify that the annexed is a true copy of the following application as filed with this Office.

Date of Application: December 17, 1999

Application Number: Japanese Patent Application No. 11-359579

Applicant: NEC Corporation

September 18, 2000

Commissioner, Patent Office

Ko2o OIKAWA

[Kind of Document]

Application for Patent

[Reference Number]

34103523

[Date of Submission]

December 17, 1999

[Addressee]

The Commissioner of the Patent Office

[IPC]

C01B 31/00

[Title of Invention]

METHOD OF PROCESSING A NANOTUBE

[Number of claims]

9

[Inventor]

[Address]

c/o NEC Corporation

7-1, Shiba 5-chome, Minato-ku, Tokyo

[Name]

Zhang YUEGANG

[Applicant]

[Identification No.]

000004237

[Name]

**NEC Corporation** 

[Attorney]

[Identification No.]

100108578

[Chartered Patent Attorney]

[Name]

Norio TAKAHASHI

[Attorney]

[Identification No.]

100064908

[Chartered Patent Attorney]

[Name]

Masatake SHIGA

[Designated Attorney]

[Identification No.]

100101465

[Chartered Patent Attorney]

[Name]

Masakazu AQYAMA

[Designated Attorney]

[Identification No.] 100108453

[Chartered Patent Attorney]

[Name] Yasuhiko MURAYAMA

[Indication of official fee]

[Account No.] 008707 [Official Fee Paid] 21000

[List of Attached Document]

[Kind of Document]Specification 1 copy[Kind of Document]Drawings 1 copy[Kind of Document]Abstract 1 copy

[Number of General Power of Attorney] 9709418

[Proofreading] Required

[Kind of Document] Specification
[Title of the prevent Invention]
METHOD OF PROCESSING A NANOTUBE
[Claim(s)]
[Claim 1]

A method of processing a nanotube comprising a first step of contacting a nanotube with a first substance and causing a reaction between the nanotube and the first substance to generate a reaction product; and

a second step of separating the nanotube from the reaction product.

[Claim 2]

A method of processing a nanotube according to the claim 1, wherein the above first step comprises a step of contacting the nanotube with the first substance before heating at least the first substance to cause a reaction between the nanotube and the first substance.

[Claim 3]

A method of processing a nanotube according to the claim 2, wherein said heating is carried out by an irradiation of a heat ray.

[Claim 4]

A method of processing a nanotube according to the claim 3, wherein said heat ray is an infrared ray.

[Claim 5]

A method of processing a nanotube according to the claim 2, wherein said heating is carried out by applying a current between said first substance and said nanotube.

[Claim 6]

A method of processing a nanotube according to the claim 1, wherein said second step comprises that said nanotube is separated from said first substance by rapidly cooling said first substance.

[Claim 7]

A method of processing a nanotube according to any one of the claims 1-6, wherein said nanotube is a single-layer winded nanotube.

[Claim 8]

A method of processing a nanotube according to any one of the claims 1-7, wherein said nanotube is a carbon nanotube.

[Claim 9]

A method of processing a nanotube according to any one of the claims 1-8, wherein said first substance is a metal or a semiconductor.

[Detail Description of Invention]

[0001]

[Field of the Invention]

The present invention relates to a method of processing a nanotube, and more particularly to a method of processing a nanotube, which is suitable for cutting the nanotube and for forming a top of the nanotube.

[0002]

[Background of the Invention]

A single-layer winded carbon nanotube has an extremely fine structure in nanometer order. Properties of the single-layer winded carbon nanotube have been on the investigation since Izima has found. The research and developments of the single-layer winded carbon nanotube for application have been active. The single-layer winded

carbon nanotube comprises a cylindrically winded single layer of graphite hexagonal network. An electron structure largely varies depending upon a diameter of the tube and a chiral angle, for which reason the electrical conductivity of the carbon nanotube varies between a conductivity of a metal and a conductivity of a semiconductor, and further the carbon nanotube exhibits a property similar to one-dimensional electric conduction. [0003]

The carbon nanotube may be applied to a field emitter. This field emitter has been known and is disclosed in (1) W.A. de Heer, A.Chatelain, and D. Ugarte, Science 270, 1179 (1995); (2) A.G. Rinzler, J.H. Hasner, P. Nikolaev, L. Lou, S.G. Kim, D. Tomanek, P. Nordlander, D.T. Colbert, and R.E. Smalley, Science, 269, 1550 (1995); (3) P.G. Collins and A. Zettl, Appl. Phys. Lett., 69, 1969 (1996); (4) Q.H. Wang, T.D. Corrigan, J.Y. Dai, P. R. H. Chang, and A.R. Krauss, Appl. Phys. Lett., 70, 3308 (1997); (5) Y. Saito, K. Hamaguchi, T. Nishino, K. hata, K. Tohji, A.Kasuya, and Y. Nishina, Jpn. J. Appl. Phys., 36, L1340 (1997); (6) J-M. Bonard, J-P. Salveiat, T. Stockli, W.A. de Heer, L. Forro, and A. Chatelain, Appl. Phys. Lett., 73, 918 (1998). [0004]

The sharpness of the top of the field emitter of the carbon nanotube emphasizes the field effect and realizes the good characteristic of the field emission.

No practicable method of operating the nanotube tip and controlling the same has been established, for which reason the conventional carbon nanotube is extremely low in probability of having an optimum directionality and serving as an useful electron emitter. As shown in (7) Y. Zhang and S. Iijima, Philos. Mag. Lett., 78, 139 (1998), the most of the manufactured single layer winded carbon nanotube is large in aspect ratio or is slender and further is curved, for which reason it is difficult to practice the single layer winded carbon nanotube. [0005]

It is actually difficult that the plural nanotube tips are arranged in the same direction and the plural nanotubes are aligned along a single line.

As shown in the above literature (7), it is actually difficult to evaluate the nanotube tip with a probe.

In order to have solved the above problem, there was proposed a method of cutting the single carbon nanotube by an oxidation using a nitric acid, an acid mixing with a nitric acid or a sulfuric acid.

[0006]

The cutting method is disclosed in (8) K.B. Shelimov, R.O. Esenaliev, A.G. Rinzler, C.B. Huffman, and R.E. Smalley, Chem. Phys. Lett., 282, 429 (1998); (9) J. Liu, M. J. Casavant, M.Cox, D.A. Walters, P. Boul, W. Lu, A.J. Rimberg, K.A. Smith, D.T. Colbert, and R.E. Smalley, Chem. Phys. Lett., 303, 125 (1999); (10) Z. Shi, Y. Lian, F. Liao, X. Zhou, Z. Gu, Y. Zhang, and S. Iijima, Solid State Comm., 112 (1999) 35.

[0007]

[Issue to be solved by the Invention]

The above cutting method for cutting the single layer winded carbon nanotube is incapable of cutting, at a specified site, the single layer winded carbon nanotube. Namely, the above cutting method is unable to specify the cutting site of the single layer winded carbon nanotube.

Since the above cutting method is the chemical wet process using the acid such as the nitric acid and the sulfuric acid, the acid provides undesirable influence to the manufacturing process. The above conventional cutting method is unsuitable for

forming the micro device. [0008]

Accordingly, it is an object of the present invention to provide a novel method of processing a nanotube free from the above problems so that it is an object of the present invention to provide a novel method of processing a nanotube without using any chemical wet process. It is a still further object of the present invention to provide a novel method of selectively processing a top portion of a nanotube in a specific shape by using a remarkably easy method. It is yet a further object of the present invention to provide a novel method of selectively processing a top portion of a nanotube in a specific shape which is suitable for an electron device such as a field emission electron gun.

[0009]

[Means for Solving the Issue]

In order to solve the above issues, the present invention provides a method of processing a nanotube as follows.

Namely, the present invention provides a method of processing a nanotube according to the claim 1, comprising a first step of contacting a nanotube with a first substance and causing a reaction between the nanotube and the first substance to generate a reaction product; and a second step of separating the nanotube from the reaction product.

[0010]

The present invention provides a method of processing a nanotube as recited in the claim 2 according to the claim 1, wherein the above first step comprises a step of contacting the nanotube with the first substance before heating at least the first substance to cause a reaction between the nanotube and the first substance.

[0011]

The present invention provides a method of processing a nanotube as recited in the claim 3 according to the claim 2, wherein said heating is carried out by an irradiation of a heat ray.

[0012]

The present invention provides a method of processing a nanotube as recited in the claim 4 according to the claim 3, wherein said heat ray is an infrared ray.

[0013]

The present invention provides a method of processing a nanotube as recited in the claim 5 according to the claim 2, wherein said heating is carried out by applying a current between said first substance and said nanotube.

[0014]

The present invention provides a method of processing a nanotube as recited in the claim 6 according to the claim 1, wherein said second step comprises that said nanotube is separated from said first substance by rapidly cooling said first substance. [0015]

The present invention provides a method of processing a nanotube as recited in the claim 7 according to any one of the claims 1-6, wherein said nanotube is a single-layer winded nanotube.

[0016]

The present invention provides a method of processing a nanotube as recited in the claim 8 according to any one of the claims 1-7, wherein said nanotube is a carbon nanotube.

[0017]

The present invention provides a method of processing a nanotube as recited in the claim 9 according to any one of the claims 1-8, wherein said first substance is a metal or a semiconductor.

The first substance is preferably used as a reaction substance reacted by contacting with said nanotube. For example, the reactive substance is Nb as the metal and Si as the semiconductor.

[0018]

[Mode for carrying out the Invention]

A first embodiment according to a method of processing a nanotube will be described in detail with reference to Fig. 1.

The present invention is applied to a single layer winded carbon nanotube (SWCNT) as an example of a nanotube. The single layer winded carbon nanotube is cut by use of Nb as a first substance to form a top of the single layer winded carbon nanotube.

[0019]

With reference to FIG. 1(a), a metal substrate, for example, a thin Nb substrate 1 was prepared as a substance which reacts with the single layer winded carbon nanotube. The Nb substrate 1 has a thickness of 2 millimeters. A hole 1a having a predetermined shape was formed by a predetermined position of the Nb substrate 1 by use of an ion milling method.

Further, single layer winded carbon nanotubes 2 were prepared by a laser ablation method.

[0020]

The single layer winded carbon nanotubes 2 were dispersed by an ultrasonic wave into an organic solvent such as ethanol, 2-propanol and acctone to form a single layer winded carbon nanotube dispersed liquid. This dispersed liquid is applied on an upper surface of the Nb substrate 1.

Next, an organic solvent is evaporated from the dispersed liquid of this Nb substrate 1. In the organic solvent, almost of the single layer winded carbon nanotubes 2 extend as single straight lines or are curved or form a bundle. After the organic solvent was evaporated, then the single layer winded carbon nanotubes 2 are kept to form the three-dimensional structure over the Nb substrate 1. Accordingly, the single layer winded carbon nanotubes 2 are in contact partially with the Nb substrate 1 at the edge of the hole 1a.

[0021]

The Nb substrate 1 with the single layer winded carbon nanotubes 2 was placed on a heat stage in a vacuum chamber of a ultra high vacuum transmission electron microscope (UHV-TEM, JEM-2000FXVII). The vacuum chamber was scaled and vacuumed to reach a vacuum degree in the range of 1.33E-7 Pa to 1.33E-6 Pa (1E-9 Torr to 1E-8 Torr). It is possible that the vacuum chamber is filled with an inert gas such as Ar gas or N2 gas at a low pressure to form an inert gas atmosphere in the vacuum chamber.

[0022]

Next, this vacuum was still kept and an infrared ray lamp was used to irradiate an infrared ray onto the Nb substrate 1 to heat the Nb substrate 1 up to a temperature. This is sufficiently high for causing a solid state reaction between the Nb substrate 1 and the single layer winded carbon nanotubes 2. For example, the highest temperature of the Nb substrate 1 may be in the range of 800-1000°C. The heat time may be in the range of 50-60 minutes.

[0023]

The solid state reaction appeared on the contact region between the Nb substrate 1 and the single layer winded carbon nanotubes 2, whereby a reaction product 3 of NbC (niobium carbide) is produced as shown in Fig. 1 (b). Next, the irradiation of the infrared ray lamp was discontinued and subsequently the sample was rapidly cooled, whereby a crack was formed at the boundary between the NbC reaction product 3 and the remaining single layer winded carbon nanotubes 2, and thus the tops of the single layer winded carbon nanotubes 2 were separated from the NbC reaction product 3. [0024]

FIG. 2 is a view of an image of the top portions of the single layer winded carbon nanotubes 2 taken by the transmission electron microscope (TEM). From FIG. 2, it can be understood that the top portions of the single layer winded carbon nanotubes 2 were selectively formed.

[0025]

In accordance with the method of processing the nanotube of the present invention, the solid state reaction is caused on the contact regions of the single layer winded carbon nanotubes 2 contacting with the Nb substrate 1 to cause these contact regions become the NbC3 reaction product, whereby the cutting portions of the single layer winded carbon nanotubes 2 could be aligned close to the contact regions with the Nb substrate 1. Therefore, non-contact portions of the single layer winded carbon nanotubes 2 structurally remain unchanged. Namely, the tops of the single layer winded carbon nanotubes 2 are formed.

[0026]

As the above mentioned, one embodiment of the method of processing the nanotube is described on the basis of the drawings, however, the concrete constructions could not be limited to the present embodiment and it could be changed on design without deviating the substance of the present invention.

For example, in the above embodiment, the Nb substrate was used as the first substance, however, other metals than Nb and semiconductors such as silicon may be available as the substance to achieve the same effects of Nb. [0027]

In the above embodiment, the carbon nanotubes were used as the nanotubes. Other nanotubes such as boron nitride (BN) as a main component based nanotubes are also available.

Not only a single layer winded nanotube but also multilayers winded nanotube such as multilayers of the carbon nanotubes are available.

In place of the infrared ray irradiation used in the above embodiment, other heating methods such as a resistance heating method of applying a current between the single layer winded carbon nanotubes 2 and the Nb substrate 1a may be available. [0028]

[Effects of the Invention]

As mentioned in the above, according to the invention, a nanotube is connected with the first substance, a first step of contacting a nanotube with a first substance and causing a reaction between the nanotube and the first substance to generate a reaction product; and a second step of separating the nanotube from the reaction product are included so that tops of the nanotube could be formed selectively by the extremely simple method.

Accordingly, the tops of the nanotube available for forming the electron elements such as an electric field effective electron gun could be formed so that it has a

remarkable effect on the electron industry. [Brief Description of Drawing]

[FIG.1]

FIG. 1 is process views illustrative of a novel method of forming a nanotube in a preferred embodiment in accordance with the present invention.
[FIG.2]

FIG. 2 is a view of an image of the top portions of the single layer winded carbon nanotubes taken by the transmission electron microscope (TEM) obtained from the method of forming a nanotube in a preferred embodiment in accordance with the present invention.

[Description of symbols]

- 1 Nb substrate
- 2 Carbon nanotubes
- 3 Nbc

[Kind of Document] Abstract

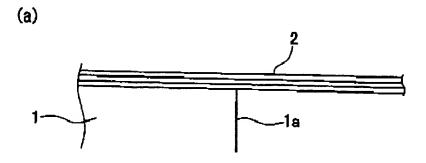
[Abstract]

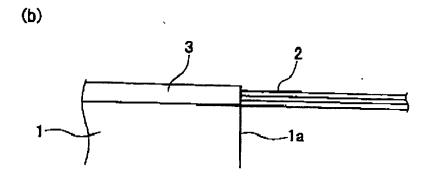
[Object] An object of the present invention is to provide a novel method of processing a nanotube which is suitable for forming a top of the nanotube with a determined form selectively by using a remarkably simple method without any chemical wet process, in particular for processing the tops of the nanotube with the form available for the electron elements such as an electric field effective electron gun.

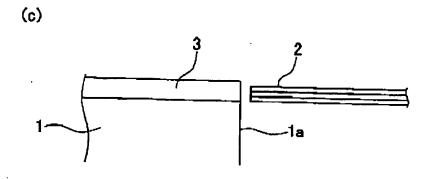
[Means] The present invention provides a method of processing a nanotube comprising a first step of contacting a nanotube 2 with a first substance 1 and causing a reaction between the nanotube 2 and the first substance 1 to generate a reaction product 3; and a second step of separating the nanotube 2 from the reaction product 3.

[Selected Drawing] FIG.1

[Title of Document] Drawings [Fig. 1]







[Fig. 2]

